

CLAIMS

We claim:

- 5 1. An apparatus for providing gases for calibration, comprising:
 at least one source that generates at least one chosen impurity in a known range of
nanograms per minute within given temperature ranges and at a predetermined flow rate;
 a supply of gas that is at least substantially pure;
 a main gas conduit;
10 a communication between said at least one impurity source and at least a portion of
said main conduit;
 a housing having an interior, wherein said impurity source and at least a portion of
said main conduit is located within the interior of said housing;
 a means for selectively raising and/or lowering the temperature of the housing interior
15 between about 60° F and about 130° F;
 a temperature controller that controls the temperature within said housing; and
 wherein said pure gas flows through said main conduit and passes by said
communication, and wherein the impurity from said source diffuses into said pure gas that
flows past said communication thereby producing a calibration gas.
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2. The apparatus of Claim 1, wherein the temperature within the housing interior is
maintained between about 60°F and about 100°F during standby or when calibration is
desired, by cooling or heating the interior as needed.
- 25 3. The apparatus of Claim 1, wherein the temperature within the housing interior is
maintained between about 75° F and about 77°F during standby or when calibration is
desired.
4. The apparatus of Claim 1, wherein the means for selectively raising and/or lowering
30 the temperature is comprised of a semi-conductor heating and cooling device.
5. The apparatus of Claim 1, further comprising a source of purge gas wherein the purge
gas flows through the main conduit and passes by said communication when the apparatus is

on standby, wherein during standby the impurity from said source diffuses into said purge gas that flows past said communication.

6. The apparatus of Claim 5, wherein the purge gas is comprised essentially of carbon dioxide, nitrogen, oxygen, helium, argon, carbon monoxide, hydrogen or a combination thereof.

7. The apparatus of Claim 4, further comprising a valve that connects the supply of pure gas and the supply of purge gas to the main conduit, and wherein the purge gas and the pure gas are introduced into the main conduit at different times.

8. The apparatus of Claim 7, further comprising a first gas conduit a second gas conduit, and a valve connecting the first and second conduits to said main conduit, wherein said pure gas is introduced into first said conduit when calibration is desired and wherein purge gas can be introduced into said second conduit when calibration gas is not needed, and wherein said valve determines which gas will flow into said main conduit.

9. The apparatus of Claim 1, further comprising at least one conduit that leads to an instrument, and wherein said calibration gas is fed to said instrument that analyzes said gas.

10. The apparatus of Claim 9, further comprising a means to connect to said apparatus to an instrument that analyzes said calibration gas.

11. The apparatus of Claim 10, wherein said instrument can be calibrated based upon results of said analysis and the known concentration of said source at a certain temperature range and flow rate.

12. The apparatus of Claim 1, wherein said housing is comprised of aluminum.

13. The apparatus of Claim 12, further comprising a fan or blower within said housing, wherein the temperature controlled air is circulated within said housing.

14. The apparatus of Claim 1, wherein the impurity source supplies at least impurity, and wherein said source generates an impurity having a concentration of about 10 nanograms/minute to about 50 nanograms/minute or higher.

15. The apparatus of Claim 1, wherein the impurity source is selected from the group consisting essentially of acetaldehyde, acetic acid, acetone, acetonitrile, acrylonitrile, ammonia, arsine, benzene, boron trichloride, boron trifluoride, 1, 3-butadiene, n-butane, t-butyl mercaptan, sec-butyl mercaptan, butyl mercaptan, carbon dioxide, carbon disulfide, carbon monoxide, carbonyl sulfide, chlorine, chloroform, di-iso propyl methyl phosphonate, di-methyl methyl phosphonate, diethyl sulfide, dimethylether, dimethyl disulfide, dimethyl sulfide, ethanol, ethyl mercaptan, ethylene oxide, formaldehyde (para), freon-11, freon-21, hydrazine, hydrogen chloride, hydrogen fluoride, hydrogen sulfide, iodine, methane, methanol, methyl chloride, methyl mercaptan, methylene chloride, nitric oxide, nitrogen dioxide, nitrogen trifluoride, nitrous oxide, oxygen, phosgene, phosphine, propane, propyl mercaptan, propylene oxide, silane, silicon tetrachloride, silicon tetrafluoride, styrene, sulfur dioxide, thiophene, toluene, vinyl acetate, vinyl chloride, water, m-xylene, o-xylene, p-xylene, or a combination thereof.

16. The apparatus of Claim 9, further comprising a third conduit, wherein at least a portion of the third conduit is located within said housing and wherein sample gas can be introduced into the third conduit.

17. The apparatus of Claim 16, wherein sample gas is conveyed through the third conduit to a calibrated instrument for analysis.

18. The apparatus of Claim 17, wherein the sample gas is selected from the group consisting essentially of carbon dioxide, nitrogen, oxygen, helium, argon, carbon monoxide, hydrogen, or a combination thereof.

19. The apparatus of Claim 1, wherein said main gas conduit is comprised of sulfur inert tubing.

20. The apparatus of Claim 16, wherein said first and third gas conduits are comprised of sulfur inert tubing.

21. The apparatus of Claim 1, wherein said apparatus holds a plurality of devices that each produces a different impurity.

22. The apparatus of Claim 1, further comprising a flow controller, wherein the flow controller allows a flow of gas between about zero liters/minute and about 2 liters/minute.

23. The apparatus of Claim 1, further comprising a pressure indicator and a back pressure regulator that monitors the pressure of gas in said main conduit and regulates the flow path of said gas.

24. The apparatus of Claim 23, further comprising means to vent gas from said main conduit if the pressure of the calibration gas drops below about 35 psi to about 40 psi .

25. The apparatus of Claim 1, further comprising an indicator that alerts an operator if the pressure of the gas in said conduit reaches an undesired pressure.

26. The apparatus of Claim 9, further comprising a solenoid valve that controls the flow of gas leading to at least one instrument.

27. An apparatus for producing calibration gases, comprising:
a source that produces a chosen gas impurity in a known range of nanograms per minute within given temperature ranges and at a predetermined flow rate;
a main gas conduit comprised of sulfur inert tubing;
a communication between said impurity source and said at least one conduit;
an insulated housing that holds said impurity source and at least a portion of the main conduit;
a semi-conductor heating and cooling device for raising and/or lowering the temperature in said housing;
a temperature controller that controls the temperature within said housing;
a means to circulate heated or cooled air within the housing;
a switch that controls the semi-conductor device and determines whether the device produces heat or cooling, wherein the temperature in said housing is maintained between about 60°F and about 130°F during standby or when calibration is desired;

wherein the purge gas flows through the conduit during standby and passes by said communication causes the impurity source to diffuse into said purge gas when the apparatus is not being used to produce a calibration gas;

a flow controller for maintaining the desired flow of gases within said conduit;

5 wherein pure gas is introduced into the conduit when calibration is desired and wherein the impurity diffuses into and mixes with the pure gas that passes by said communication to provide a calibration gas; and

wherein said calibration gas is conveyed through the conduit to an instrument.

10 28. The apparatus of Claim 27, further comprising a first and a second gas conduit, wherein said first conduit is connected to a source of pure gas and wherein said second conduit is connected to a source of purge gas, and wherein said first and second conduits are connected to a main conduit by a valve that determines which gas will flow into said main conduit.

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29. The apparatus of Claim 27, further comprising fittings, wherein the internals of said fittings are coated with sulfur inert coating to prevent permeation of sulfur into said fittings.

20 30. The apparatus of Claim 27, wherein said impurity source is placed in a header or manifold that is located within said housing and that communicates with said conduit.

31. The apparatus of Claim 30, further comprising a plurality of headers, wherein each header holds a different source.

25 32. The apparatus of Claim 27, wherein the switch for the semi-conductor device is manually controlled and/or automatically controlled.

33. The apparatus of Claim 27, wherein the housing is comprised of aluminum and is insulated.

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34. The apparatus of Claim 27, wherein the impurity source produces at least one impurity of a known concentration of about 10 to about 50 nanograms per minute or higher that is measurable in ppm or ppb.

35. The apparatus of Claim 27, which is contained within a portable case.

36. The apparatus of Claim 27, wherein a 12 volt direct current power supply is used to operate said apparatus.

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37. A method of producing gases for calibration, comprising:

providing a source for generating a chosen impurity at a rate having a known value range of nanograms per minute within given temperature ranges and a predetermined flow rate;

10 supplying at least one gas conduit;

providing an insulated housing;

placing the impurity source and at least a portion of said at least one gas conduit within said housing;

15 controlling the temperature within said housing at between about 60°F and about 130°F during standby or when calibration is desired;

providing a communication between the said conduit and said impurity source; and providing a supply of pure gas;

20 making a calibration gas by and allowing the impurity source to diffuse into said pure gas that flows past said communication, thereby mixing with said pure gas; and using the calibration gas to calibrate an instrument.

38. The method of Claim 37, further comprising the step of using a semi-conductor device to selectively raise and/or lower the temperature within said housing between about 60° F and about 130° F to create the optimal temperature for the chosen impurity source.

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39. The method of Claim 37, further comprising the steps of:

providing a supply of purge gas;

introducing purge gas into said at least one conduit, wherein purge gas flows through said conduit during standby and causes the impurity source to diffuse into said purge gas that flows past said communication; and

30 changing the purge gas to pure gas when a calibration gas is desired.

40. The method of Claim 37, further comprising the steps of:
analyzing the calibration gas in an instrument; and
calibrating an instrument based upon the analysis and the known value of said
impurity source.

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41. The method of Claim 37, wherein said impurity source produces at least one impurity
of a known concentration of about 10 to about 50 nanograms per minute that is measurable in
ppm, ppb, or ppt.

10 42. The method of Claim 40, further comprising the step of providing a sample gas
comprising an unknown amount and/or type of impurities for analysis.

43. The method of Claim 42, further comprising the step of analyzing said sample gas in
said calibrated instrument.

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44. The method of Claim 43, further comprising the step of comparing the results of the
sample analysis with known results to determine the type and amount of impurities in said
sample.

20 45. The method of Claim 37, further comprising the step of making at least a portion of
said at least one conduit from tubing that has a sulfur inert interior.

46. The method of Claim 40, wherein said instrument comprises a sulfur analyzer and/or
a gas chromatograph.

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47. The method of Claim 39, wherein the purge gas and the pure gas are introduced into
the same conduit at different times.

30 48. The method of Claim 42, further comprising the step of supplying at least one
additional conduit for a sample gas, wherein said additional conduit leads to the instrument.

49. The method of Claim 37, further comprising the steps of:

providing a first conduit, a second conduit, and a valve connecting said first and second conduits to said at least one conduit, wherein said at least one conduit is the main gas conduit;

5 providing a supply of purge gas;

connecting said supply of pure gas to said first conduit;

connecting said supply of purge gas to said second conduit;

running purge gas through said second conduit during standby and opening said valve so that purge gas is introduced into the main conduit and flows past said communication; and

10 changing said valve to stop the flow of purge gas into said main conduit and allowing pure gas to flow through said first conduit into said main conduit when calibration is desired.

50. The method of Claim 37, wherein the impurity source supplies at least one impurity and wherein said impurity is selected from the group consisting essentially of acetaldehyde,

15 acetic acid, acetone, acetonitrile, acrylonitrile, ammonia, arsine, benzene, boron trichloride, boron trifluoride, 1, 3-butadiene, n-butane, t-butyl mercaptan, sec-butyl mercaptan, butyl mercaptan, carbon dioxide, carbon disulfide, carbon monoxide, carbonyl sulfide, chlorine, chloroform, di-isopropyl methyl phosphonate, di-methyl methyl phosphonate, diethyl sulfide, dimethylether, dimethyl disulfide, dimethyl sulfide, ethanol, ethyl mercaptan,
20 ethylene oxide, formaldehyde (para), freon-11, freon-21, hydrazine, hydrogen chloride, hydrogen fluoride, hydrogen sulfide, iodine, methane, methanol, methyl chloride, methyl mercaptan, methylene chloride, nitric oxide, nitrogen dioxide, nitrogen trifluoride, nitrous oxide, oxygen, phosgene, phosphine, propane, propyl mercaptan, propylene oxide, silane, silicon tetrachloride, silicon tetrafluoride, styrene, sulfur dioxide, thiophene, toluene, vinyl
25 acetate, vinyl chloride, water, m-xylene, o-xylene, p-xylene, or a combination thereof.

51. The method of Claim 37, further comprising the step of using a flow controller to control the flow of gas inside said at least one conduit between about zero liters per minute to about two liters per minute.

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52. The method of Claim 37, further comprising the step of providing a vent, wherein gas of an undesired pressure is vented before it reaches said instrument.

53. The method of Claim 37, further comprising the step of using a rotometer to ensure a steady gas flow to said instrument.

54. A system for providing a more accurate calibration of instruments for testing gases,
5 comprising:

at least one source that generates at least one chosen impurity in a known range of nanograms per minute at a known temperature range and at a predetermined flow rate

a supply of pure gas;

a main gas conduit;

10 a housing having an interior that holds the source of said at least one purity and at least a portion of said main conduit;

a means for selectively raising and/or lowering the temperature of the housing interior between about 60° F and 130° F, wherein the temperature of said source and said at least a portion of said main conduit is selectively raised and/or lowered between about 60° F and
15 about 130° F;

a flow controller that allows gas to flow through said main conduit between about 0.5 liters/minute and about 2 liters/minute;

a communication between said impurity source and said main conduit, wherein pure gas flows through the main conduit and past said communication wherein said impurity
20 source diffuses into the pure gas and mixes with said pure gas to form a calibration gas;

an instrument that is capable of being calibrated;

a connection between said instrument and said main conduit, wherein said calibration gas is fed to the instrument that analyzes said gas; and

wherein said instrument is calibrated based said analysis and the known value of said
25 source.

55. The system of Claim 54, further comprising:

a supply of purge gas;

a valve, wherein said valve determines whether said pure and/or purge flows into said
30 main gas conduit; and

wherein purge gas is run through main conduit during standby and pure gas is run through said main conduit when calibration is desired.

56. The system of Claim 54, further comprising:

a first gas conduit, a second gas conduit, and a valve connecting the first and second conduits to said main conduit, wherein said valve determines which gas will flow into said main conduit

5 a supply of purge gas, wherein purge gas flows through said second gas conduit, and wherein pure gas flows through said first gas conduit;

wherein said pure gas is introduced into first said conduit when calibration is desired and wherein purge gas can be introduced into said second conduit when calibration gas is not needed, wherein either pure gas or purge gas can be introduced into the main conduit to flow
10 past said communication; and

wherein said valve is changed to stop the flow of purge gas into said main conduit and pure gas is allowed to flow through said first conduit into said main conduit when calibration is desired.

15 57. The system of Claim 54, wherein said main conduit is comprised of sulfur inert tubing.

58. The system of Claim 54 wherein the means for selectively raising and/or lowering the temperature is comprised of a semi-conductor heating and cooling device.

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59. The system of Claim 56, wherein purge gas flows through said main conduit during standby and the impurity from said source diffuses into said purge gas.

60. The system of Claim 54, wherein the purge gas is selected from the group consisting
25 essentially of carbon dioxide, nitrogen, oxygen, helium, argon, carbon monoxide, hydrogen, or a combination thereof.

61. The system of Claim 54, wherein said impurity source produces at least one impurity having a known concentration of about 10 to about 50 nanograms per minute that is
30 measurable in ppm, ppb, or ppt.

62. The apparatus of Claim 61, used in conjunction with an instrument to be calibrated, wherein said instrument analyzes said calibration gas.

63. The system of Claim 62, wherein said instrument is calibrated based upon said analysis and the known value of said calibration gas.

64. The system of Claim 63, further comprising a gas sample comprising an unknown amount of impurity for analysis, wherein said sample is analyzed by said calibrated instrument.

65. The system of Claim 64, wherein said sample analysis yields a result, and wherein said result of the analysis is compared with known results to determine the type and amount of impurities in said sample.

66. The system of Claim 62, wherein said instrument comprises a gas chromatograph.

67. The system of Claim 62, wherein said instrument comprises a sulfur analyzer.

68. The system of Claim 62, wherein said instrument comprises a gas monitoring system or a hydrocarbon analyzer.

69. The system of Claim 66, further comprising a rotometer between the main conduit and said gas chromatograph.